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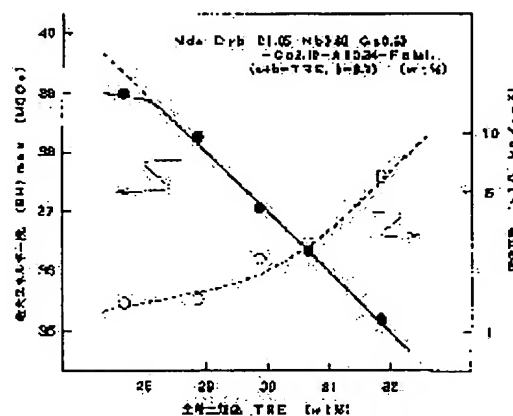
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(54) ND-FE-CO-B TYPE SINTERED MAGNET

(57)Abstract:

PURPOSE: To obtain a Nd-Fe-Co-B type sintered magnet excellent in corrosion resistance and heat resistance.

CONSTITUTION: A Nd-Fe-Co-B type sintered magnet which comprises 28 to 32wt.% of R (where R is at least one selected from Y and rare earth elements, and 3.0 to 8.0wt.% of R is Dy and the remainder of R is one or two of Nd and Pr, Nd being contained in R by 50 at% or more), 0.1 to 1.0wt.% of Al, 0.5 to 2.0wt.% of B, 0.1 to 2.0wt.% of Nb, and unavoidable impurities, with the remainder being composed of a composition mainly consisting of Fe. In addition, having a coercive force iH_c of 20kOe or greater and a maximum magnetic energy product (BH) max of 30MGOe or greater, it is excellent in corrosion resistance and heat resistance.

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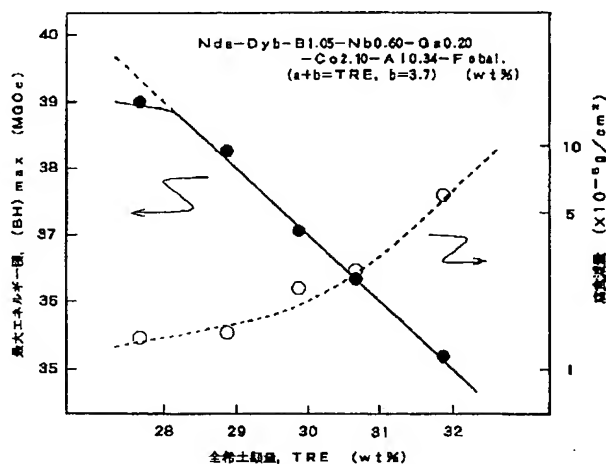
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(54)【発明の名称】 Nd-Fe-Co-B型焼結磁石

(57)【要約】

【目的】 耐食性、耐熱性に優れたNd-Fe-Co-B型焼結磁石を提供する。

【構成】 28～32wt%のR（但し、RはY及び希土類元素から選ばれた少なくとも1種であり、Rの内、3.0～8.0wt%がDy、残りは、Nd又はPrの1種又は2種であり、NdをRの内に50at%以上を含む）、5.0wt%以下のCo（但し、Coは必ず含む）、0.1～1.0wt%のAl、0.5～2.0wt%のB、0.1～2.0wt%のNb、0.05～1.0wt%のGa、1000ppm～6000ppmの酸素、及び不可避免の不純物を含有し、残部が主としてFeからなり保磁力*i*H_cが20kOe以上、最大磁気エネルギー積(BH)_{max}が30MGOe以上である耐食性、耐熱性に優れたNd-Fe-Co-B型焼結磁石。



【特許請求の範囲】

【請求項1】 28～32wt%のR（但し、RはY及び希土類元素から選ばれた少なくとも1種であり、Rの内、3.0～8.0wt%がDy、残りは、Nd又はPrの1種又は2種であり、NdをRの内に50at%以上を含む）、5.0wt%以下のCo（但し、Coは必ず含む）、0.1～1.0wt%のAl、0.5～2.0wt%のB、0.1～2.0wt%のNb、0.05～1.0wt%のGa、1000ppm～6000ppmの酸素、及び不可避免的不純物を含有し、残部が主としてFeからなり保磁力*i*Hcが20kOe以上、最大磁気エネルギー積（BH）*max*が30MGOe以上である耐食性、耐熱性に優れたNd-Fe-Co-B型焼結磁石。

【請求項2】 Ga含有量が0.05～0.8wt%である請求項1記載のNd-Fe-Co-B型焼結磁石。

【請求項3】 Ga含有量が0.1～0.6wt%である請求項1記載のNd-Fe-Co-B型焼結磁石。

【請求項4】 Ga含有量が0.1～0.4wt%である請求項1記載のNd-Fe-Co-B型焼結磁石。

【請求項5】 Rのうち3.0～5.0wt%がDyであり、最大磁気エネルギー積（BH）*max*が35MGOe以上である請求項1～請求項4のいずれかに記載のNd-Fe-Co-B型焼結磁石。

【請求項6】 Rのうち5.0～8.0wt%がDyであり、保磁力*i*Hcが25kOe以上である請求項1～請求項4のいずれかに記載のNd-Fe-Co-B型焼結磁石。

【請求項7】 常温磁気特性として
 $iHc \geq 20 \text{ kOe}$ 、 $(BH)_{\max} \geq 30 \text{ MGOe}$ であり、23℃から120℃の残留磁束密度*B*_r、保磁力*i*Hc各々の温度係数 α 、 β が
 $-0.12 \leq \alpha \leq -0.08 \text{ \%}/^\circ\text{C}$
 $-0.65 \leq \beta \leq -0.40 \text{ \%}/^\circ\text{C}$
 である請求項1記載のNd-Fe-Co-B型焼結磁石。

【請求項8】 表面にNiメッキを施した請求項1～請求項7のいずれかに記載のNd-Fe-Co-B型焼結磁石。

【発明の詳細な説明】

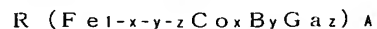
【0001】

【産業上の利用分野】本発明はネオジウム（Nd）、鉄（Fe）、コバルト（Co）及びホウ素（B）を主成分とする永久磁石に関し、特に優れた耐食性、耐熱性を有するNd-Fe-Co-B型焼結永久磁石に関するものである。

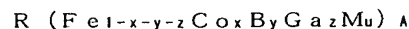
【0002】

【従来の技術】Nd-Fe-B型焼結磁石及びNd-Fe-Co-B型焼結磁石は、SmCo₅型焼結磁石或いはSm₂Co₁₇型焼結磁石と比較して高いエネルギー積

（BH）*max*を有するので、種々の用途に使用されるようになっている。しかしながら、Nd-Fe-B型焼結磁石及びNd-Fe-Co-B型焼結磁石は、これらSm-Co型焼結磁石と比較して熱安定性に劣るので、その熱安定性を増す為に種々の試みが提案されている。特開昭64-7503号公報には、熱安定性の良好な永久磁石として一般式：



（但し、Rは希土類元素から選ばれた少なくとも1種であり、 $0 \leq x \leq 0.7$ 、 $0.02 \leq y \leq 0.3$ 、 $0.01 \leq z \leq 0.15$ 、 $4.0 \leq A \leq 7.5$ である。）及び、



（但し、Rは希土類元素から選ばれた少なくとも1種であり、MはNb、W、V、Ta及びMoから選ばれた1種または2種以上の元素であり、 $0 \leq x \leq 0.7$ 、 $0.02 \leq y \leq 0.3$ 、 $0.001 \leq z \leq 0.15$ 、 $u \leq 0.1$ 、 $4.0 \leq A \leq 7.5$ である。）により表されるものを開示している。

【0003】

【発明が解決しようとする課題】しかしながら、高耐食性、高耐熱特性を有し、かつ同時に高い水準の保磁力*i*Hc、エネルギー積（BH）*max*を兼ね備えたNd-Fe-Co-B型異方性焼結磁石を安定的に生産しようとする場合には前記公知技術を超えて更に詳細な研究・検討に基づいた成分組成範囲の限定、酸化物の限定等が必要であることが分かった。本発明はこのような知見に基づき、特に耐食性、耐熱性に優れたNd-Fe-Co-B型焼結磁石を提供するものである。本発明は、Co、Dy、Nbを有効に利用することによって耐食性を著しく高め、Dy量、Ga量を特定範囲とすることにより高耐熱性を付与し、同時に希土類R量を低め、かつ、酸素量を限定することにより保磁力*i*Hcが大きく、かつエネルギー積（BH）*max*の大きいNd-Fe-Co-B型焼結磁石を安定的に提供するものである。

【0004】

【課題を解決するための手段】本発明は、28～32wt%のR（但し、RはY及び希土類元素から選ばれた少なくとも1種であり、Rの内、3.0～8.0wt%がDy、残りは、Nd又はPrの1種又は2種であり、NdをRの内に50at%以上を含む）、5.0wt%以下のCo（但し、Coは必ず含む）、0.1～1.0wt%のAl、0.5～2.0wt%のB、0.1～2.0wt%のNb、0.05～1.0wt%のGa、1000ppm～6000ppmの酸素、及び不可避免的不純物を含有し、残部が主としてFeからなり保磁力*i*Hcが20kOe以上、最大磁気エネルギー積（BH）*max*が30MGOe以上である耐食性、耐熱性に優れたNd-Fe-Co-B型焼結磁石である。本発明の永久磁石の組成の限定理由について、以下詳細に説明す

る。

【0005】本発明においてRは28～32wt%の範囲で含有される。後述の実施例4に示されるようにR量が32wt%以下と少ないほど(BH)max、および耐食性の向上に有効である。しかし、28wt%未満ではインゴット中に α -Feが発生し易くなり(BH)maxの増大は期待しにくい。よってR量は28～32wt%とする。RはNdを主体とするために、R成分の内、50at%以上のNdを含有するものとする。Rの内には3.0～8.0wt%のDyを含有するが、残部はNd単独又はNdとPrとの混合である。Prは保磁力iHcの向上に効果がある。

【0006】DyをR成分として含有させることによって、キュリー点Tcが上昇するとともに異方性磁場(HA)が増大して保磁力iHcが向上し、耐熱性を著しく向上させる。また、Dyは耐食性向上にも効果がある。本発明において、Dyの含有量が3.0wt%より少ないと、熱安定性、耐食性を向上させるという本発明の目的は達成されない。しかし、8.0wt%よりも含有量が多くなると、残留磁束密度Br及び最大エネルギー積(BH)maxの低下による磁気特性の劣化が著しい。したがって、Dyの含有量は3.0～8.0wt%とする。Dyが5.0wt%よりも含有量が多いと、残留磁束密度Br及び最大エネルギー積(BH)maxの低下はあるものの、25kOe以上の保磁力iHcを得ることができる。よってより高保磁力特性を得ようとする場合にはDyの含有量を5.0～8.0wt%とする。逆に大きな残留磁束密度Br及び最大エネルギー積(BH)maxを得ようとする場合にはDyの含有量を3.0～5.0wt%とすればよい。

【0007】本発明においてCoは、残留磁束密度Brを殆ど低下させることなく磁石合金自身の耐食性を改善するとともに耐食コーティングであるNiメッキの密着性を向上することにより耐食性を向上させる効果がある。また、主相(Nd₂Fe₁₄B)中のFeがCoに置換されることによりキュリー点Tcを上昇させる効果もある。しかしながらCoの置換量を多くすると、焼結時の異常粒成長を原因とする粗大結晶粒が発生し、保磁力iHc及びヒステリシスカーブの角型性が低下する。したがってCo含有量は5.0wt%以下とする。

【0008】本発明においてAlは、Co添加材の熱処理時の温度条件を緩和する効果がある。すなわち、Coを含有すると材料は熱処理温度の変動に対して磁気特性や熱安定性の変動が大きい。そこに適量のAlを添加すると、熱処理条件が多少変動しても磁気特性や熱安定性が変動しなくなる。これにより、永久磁石の生産管理が容易となり、品質の安定した永久磁石を効率よく生産できるようになる。Alの含有量が0.1wt%未満では上記の効果は不十分である。一方、1.0wt%を超えると、残留磁束密度Brの低下が顕著になる。従ってA

lの含有量は0.1～1.0wt%とする。Bは、0.5wt%未満の場合には高保磁力が得られず、一方、2.0wt%を超えると、Bに富む非磁性相が増加し、残留磁束密度Brが低下する。そのため、0.5～2.0wt%とする。好ましいBの含有量は0.8～1.2wt%である。

【0009】Gaは、残留磁束密度Brを殆ど低下させず、保磁力iHcを向上する効果がある。Ga含有量が0.05wt%未満の場合は保磁力iHcを向上する効果が十分でない。Ga含有量が1.0wt%を超えると、残留磁束密度Brが低下し、所望の高エネルギー積が得られない。よって、Ga含有量は0.05～1.0wt%とする。Ga含有量が多いと磁石のヒステリシスカーブの角型性が悪くなるので、高い角型性を付与するためにも好ましいGaの含有量は0.05～0.8wt%である。より好ましいGaの含有量は0.1～0.6wt%である。更に好ましくは0.1～0.4wt%である。

【0010】本発明の永久磁石は、上記成分の他に0.1～2.0wt%のNbを含有する。Nbは焼結時に結晶粒が粗大化することを抑制する効果がある。この効果により、保磁力iHcが向上し、ヒステリシスカーブの角型性が良好になる。また、焼結体の結晶粒が微細になることは磁石の良好な着磁性に大きく寄与し、さらに着磁性の良好なNd-Fe-Co-B型焼結磁石は優れた耐熱性を有する。よって、耐熱性を有する磁石にNbは有効な添加物である。Nbの含有量が0.1wt%未満の場合、粗大粒を抑制する効果が不十分である。一方、Nbの含有量が2.0wt%を超える場合には、NbもしくはNb-Feの非磁性ホウ化物が多く発生し、残留磁束密度Br及びキュリー点Tcが著しく低下し好ましくない。よって、Nbの含有量は0.1～2.0wt%とする。好ましくは、0.1～1.0wt%である。

【0011】酸素含有量は、1000ppm～6000ppmとする。酸素が1000ppmより少ない場合には磁石粉、及びその圧密体が発火しやすく工業生産上危険がある。一方、6000ppmより多い場合には酸素が希土類R成分と反応して希土類酸化物を形成し、高保磁力及び高エネルギー積の磁石を得るのが困難になる。

【0012】本発明の焼結磁石は、次のようにして製造することができる。即ち、一定の成分組成を有するインゴットを真空溶解で製作し、次にこのインゴットを粗粉砕することにより粒径500μm程度の粗粉を得る。この粗粉をジェットミルを用い、不活性ガス雰囲気中で微粉砕し平均粒径3.0～6.0μm(F. S. S. S.)の微粉を得る。次にこの微粉を配向磁場15kOe、成形圧力1.5ton/cm²の条件下で磁場中プレス成形後、1000～1150℃の温度範囲で焼結する。焼結後の熱処理は、次のように行なうことができる。成形体を焼結して得た焼結体をいったん室温まで冷却す

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る。焼結後の冷却速度は最終製品の保磁力 iH_c に殆ど影響を与えない。次いで、 $800 \sim 1000^\circ\text{C}$ の温度に加熱し、 $0.2 \sim 5$ 時間保持する。これを第1次熱処理とする。加熱温度が 800°C 未満または 1000°C を超える場合、充分な高保磁力が得られない。加熱保持の後で $0.3 \sim 50^\circ\text{C}/\text{分}$ の冷却速度で室温ないし 600°C の温度まで冷却する。冷却速度が $50^\circ\text{C}/\text{分}$ を超える場合は、時効のために必要な平衡相が得られず、充分な高保磁力が得られない。また、 $0.3^\circ\text{C}/\text{分}$ 未満の冷却速度は熱処理に時間を要し、工業生産上経済的でない。好ましくは、 $0.6 \sim 2.0^\circ\text{C}/\text{分}$ の冷却速度が選ばれる。冷却終了温度は室温が望ましいが、多少保磁力 iH_c を犠牲にすれば 600°C までとし、その温度以下は急冷してもよい。好ましくは、常温 $\sim 400^\circ\text{C}$ の温度まで冷却する。熱処理は更に $500 \sim 650^\circ\text{C}$ の温度で $0.2 \sim 3$ 時間行う。これを第2次熱処理とする。組成によって異なるが、好ましくは $540 \sim 640^\circ\text{C}$ での熱処理が有効である。熱処理温度が 500°C 未満の場合及び 650°C より高い場合は、高保磁力が得られても不可逆減磁率の低下がおきる。熱処理後は第1次熱処理と同様、 $0.3 \sim 400^\circ\text{C}/\text{分}$ の冷却速度で冷却する。冷却は水中、シリコンオイル中、アルゴン気流中等で行うことができる。冷却速度が $400^\circ\text{C}/\text{分}$ を越える場合、急冷により試料に亀裂が入り、工業的に価値のある永久磁石材料が得られない。また、 $0.3^\circ\text{C}/\text{分}$ 未満の場合、冷却過程で保磁力 iH_c に好ましくない相が出現する。

【0013】

【実施例】以下、実施例により本発明を更に詳細に説明する。

(実施例1) 金属Nd、金属Dy、Fe、Co、ferro-B、ferro-Nb、金属Gaを所定の重量秤量し、これを真空溶解して重量10kgのインゴットを作製した。このインゴットの成分分析を行なうと重量比で以下のような組成であった。Nd27.5-Dy3.6-B1.03-Nb0.58-Ga0.18-Co2.02-A10.35-Fe bal. (wt%)

このインゴットをハンマーで解砕した後、さらに粗粉碎機を用い不活性ガス雰囲気中での粗粉碎を行い $500 \mu\text{m}$ 以下の粒度の粗粉を得た。この粗粉を同じくジェットミルを用い不活性ガス雰囲気中で微粉碎をして微粉を得た。この微粉は平均粒径 $4.0 \mu\text{m}$ (F. S. S.) であり、含有酸素量が 5500 ppm であった。次に、この微粉を配向磁場強度 15 kOe 、成形圧力 $1.5 \text{ ton}/\text{cm}^2$ の条件下で磁場中プレス成形し、 $30 \times 20 \times 15$ の成形体を作製した。この成形体は実質的に真空の条件で $1080^\circ\text{C} \times 3 \text{ hr}$ の焼結を行い、得られた焼結体に $900^\circ\text{C} \times 2 \text{ hr}$ の第1次熱処理、次いで $530^\circ\text{C} \times 2 \text{ hr}$ の第2次熱処理を施した。得られた焼結体の密度は $7.55 \text{ g}/\text{cc}$ 、また含有酸素量は 4800 ppm であった。この試料の常温磁気特性を測

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定したところ以下の様な値を得た。

$$Br = 12.6 \text{ kG}$$

$$bH_c = 11.6 \text{ kOe}$$

$$iH_c = 21.8 \text{ kOe}$$

$$(BH)_{\max} = 35.6 \text{ MGOe}$$

更にキュリー点 T_c として 340°C 、 23°C から 120°C の Br と iH_c の温度係数 α 、 β として各々 -0.10 、 $-0.52\%/^\circ\text{C}$ の値を得た。またパーミアンス係数 $P_c = 1.0$ 、 2.0 形状をした試料の 100°C での不可逆減磁率は各々 2.1 、 1.1% であり優れた耐熱性を有している。

【0014】(実施例2) 実験条件を変えて、実施例1と同様にして次の実験結果を得た。

組成 : Nd25.5-Dy6.4-B1.04-Nb0.55-Ga0.22-Co2.00-A10.36-Fe bal. (wt%)

焼結 : $1100^\circ\text{C} \times 2 \text{ hr}$

第1次熱処理 : $900^\circ\text{C} \times 2 \text{ hr}$

第2次熱処理 : $530^\circ\text{C} \times 2 \text{ hr}$

常温磁気特性 : $Br = 11.4 \text{ kG}$

$$bH_c = 11.0 \text{ kOe}$$

$$iH_c = 27.8 \text{ kOe}$$

$$(BH)_{\max} = 31.3 \text{ MGOe}$$

キュリー点 : $T_c = 340^\circ\text{C}$

不可逆減磁率 [at 100°C] : $P_c = 1.0 \rightarrow 1.8\%$

$P_c = 2.0 \rightarrow 0.8\%$

Br 温度係数 (α)、 iH_c 温度係数 (β) [$23^\circ\text{C} \sim 120^\circ\text{C}$] : $\alpha = -0.09\%/^\circ\text{C}$

$\beta = -0.51\%/^\circ\text{C}$

焼結体含有酸素量 : 5800 ppm

実施例1同様、常温磁気特性と共に高温特性に優れており、耐熱性に優れた磁石を得ることができる。

【0015】(実施例3) ジジムタル (Nd70wt%-Pr30wt%) を使用し、実施例1、2と同様にして次の実験結果を得た。

組成 : Nd18.9-Pr5.1-Dy7.3-B1.10-Nb0.71-Ga0.37-Co4.72-A10.33-Fe bal. (wt%)

焼結 : $1080^\circ\text{C} \times 2 \text{ hr}$

第1次熱処理 : $900^\circ\text{C} \times 2 \text{ hr}$

第2次熱処理 : $520^\circ\text{C} \times 2 \text{ hr}$

常温磁気特性 : $Br = 11.5 \text{ kG}$

$$bH_c = 10.9 \text{ kOe}$$

$$iH_c = 30.0 \text{ kOe}$$

$$(BH)_{\max} = 31.2 \text{ MGOe}$$

キュリー点 : $T_c = 375^\circ\text{C}$

不可逆減磁率 [at 100°C] : $P_c = 1.0 \rightarrow 1.4\%$

$P_c = 2.0 \rightarrow 0.5\%$

Br 温度係数 (α)、 iH_c 温度係数 (β) [23°C

～120℃] : $\alpha = -0.09\%/^{\circ}\text{C}$

$\beta = -0.48\%/^{\circ}\text{C}$

焼結体含有酸素量 : 5400ppm

ジジメタルを用いた場合でも、実施例1、2と同様常温磁気特性、高温特性、耐熱性に優れた磁石を得ることができる。

【0016】(実施例4) 金属Nd、金属Dy、Fe、Co、ferro-B、ferro-Nb、金属Gaを所定の重量秤量し、これを真空溶解して重量各10kgのインゴットを作製した。このインゴットの成分分析を行なうと重量比で以下のような組成であった。

$\text{Nd a-Dy b-B1.05-Nb0.58-Ga0.20}$
 $-Co0.20-A10.33-Fe bal.$

($a+b=TRE$, $b=3.7$) (wt%)

各々のインゴットをハンマーで解砕した後、さらに粗粉砕機を用い不活性ガス雰囲気中での粗粉砕を行い500 μm 以下の粒度の粗粉を得た。この粗粉を同じくジェットミルを用い不活性ガス雰囲気中で微粉砕をして微粉を得た。この微粉は平均粒径3.7 μm (F. S. S. S.)であり、含有酸素量は1500～5000ppmであった。次に、この微粉を配向磁場強度15kOe、成形圧力1.5ton/cm²の条件下で磁場中プレス成形し、30×20×15の成形体を作製した。この成形体は実質的に真空の条件で1070℃×2hrの焼結を行い、得られた焼結体に900℃×2hrの第1次熱処理、次いで540℃×2hrの第2次熱処理を施した。得られた焼結体の密度は7.55～7.58g/cc、また含有酸素量は1000～4000ppmであった。この試料について、TRE含有量に対して最大エネルギー積(BH)_{max}及び腐食減量がどのように変化するかを測定し、図1に示すような結果を得た。腐食減量は磁石を温度120℃、湿度90%、気圧1.0atmの環境中に100時間暴露したときに得られたものである。図1に示されるようにTRE量を少なくすることによって(BH)_{max}を向上することができるが、28wt%未満とするとインゴット中に $\alpha\text{-Fe}$ が発生し易くなり(BH)_{max}の増大は期待しにくい。腐食減量もやはりTRE量を少なくすることによって減少させることができる。これは、TREを少なくすることによって腐食しやすいNd-rich相が減少する為である。しかしながら、TRE量を28～32wt%という低い値としても含有酸素量が6000ppmを超えてしまうと保磁力iHcの減少が著しくなるため、酸素量は1000～6000ppmとする。図2に焼結磁石中の酸素含有量と磁気特性の関係を示す。

【0017】(実施例5) 金属Nd、金属Dy、Fe、Co、ferro-B、ferro-Nb、金属Gaを*

(Nd+Pr) 28.1-Dy3.6-B1.03-Nb0.58-Ga b

-Co2.05-A10.35-Fe bal. ($0 \leq b \leq 0.6$) (wt%)

各々のインゴットをハンマーで解砕した後、さらに粗粉

*所定の重量秤量し、これを真空溶解して重量各10kgのインゴットを作製した。このインゴットの成分分析を行なうと重量比で以下のような組成であった。

$\text{Nd30.5-a-Dy a-B1.03-Nb0.59-Ga b}$
 $-Co2.10-A10.34-Fe bal.$

($2.8 \leq a \leq 8.5$, $0 \leq b \leq 1.2$) (wt%)

各々のインゴットをハンマーで解砕した後、さらに粗粉砕機を用い不活性ガス雰囲気中での粗粉砕を行い500 μm 以下の粒度の粗粉を得た。この粗粉を同じくジェットミルを用い不活性ガス雰囲気中で微粉砕をして微粉を得た。この微粉は平均粒径3.8 μm (F. S. S. S.)であり、含有酸素量は5500～6400ppmであった。次に、この微粉を配向磁場強度15kOe、成形圧力1.5ton/cm²の条件下で磁場中プレス成形し、30×20×15の成形体を作製した。この成形体は実質的に真空の条件で1100℃×2hrの焼結を行い、得られた焼結体に900℃×2hrの第1次熱処理、次いで580℃×2hrの第2次熱処理を施した。得られた焼結体の密度は7.55～7.59g/cc、また含有酸素量は5000～5900ppmであった。これら試料について、常温磁気特性を測定し、図3、図4及び図5に示すような結果を得た。図3はDy=5.0wt%として測定した結果であるがGa含有量が0.05wt%未満では効果を発揮しにくい、一方、1.0wt%以上にしても最大エネルギー積(BH)_{max}の減少が著しくなるだけで保磁力iHcの向上はさほど期待できないので0.05～1.0wt%が適量である。GaはDyに比較して(BH)_{max}を著しく低下することなく保磁力iHcを向上させる効果が大きいので0.01～1.0wt%の含有は本発明において必須となる。図4にはGa含有量を0.20wt%としてDy含有量を変化させた結果を示す。Dy含有はiHcの向上に大きく貢献するが一方で(BH)_{max}を著しく低下させるので含有量は3.6～8.0wt%が適量である。図5にはDy含有量をパラメータとしてGa含有量を0～0.6wt%まで変化させた場合の結果を示すが、Dy含有量が8.0wt%を超えると(BH)_{max}が著しく低下する。また、Dy含有量が3.0wt%未満であると、20kOeを越える高保磁力が得にくい。

【0018】(実施例6) ジジメタル(Nd70wt%-Pr30wt%)、金属Dy、Fe、Co、ferro-B、ferro-Nb、金属Gaを所定の重量秤量し、これを真空溶解して重量各10kgのインゴットを作製した。このインゴットの成分分析を行なうと重量比で以下のような組成であった。

μm 以下の粒度の粗粉を得た。この粗粉を同じくジェットミルを用い不活性ガス雰囲気中で微粉砕をして微粉を得た。この微粉は平均粒径 $3.7\mu\text{m}$ (F. S. S. S.) であり、含有酸素量は 5600ppm であった。次に、この微粉を配向磁場強度 15kOe 、成形圧力 1.5ton/cm^2 の条件下で磁場中プレス成形し、 $30\times 20\times 15$ の成形体を作製した。この成形体は実質的に真空の条件で $1080^\circ\text{C}\times 2.5\text{hr}$ の焼結を行い、得られた焼結体に $890^\circ\text{C}\times 2\text{hr}$ の第1次熱処理、次いで $530^\circ\text{C}\times 2\text{hr}$ の第2次熱処理を施した。得られた焼結体の密度は $7.55\sim 7.58\text{g/cc}$ 、また含有酸素量は 2800ppm であった。これら試料について、常温磁気特性を測定し、図6に示すような結

(Nd+Pr) 28.0-Dy 4.0-B1.03-Nbx-Ga 0.15

-Co 2.04-Al 0.35-Fe bal. ($0\leq x\leq 1.0$) (wt %)

各々のインゴットをハンマーで解砕した後、さらに粗粉砕機を用い不活性ガス雰囲気中で粗粉砕を行い $500\mu\text{m}$ 以下の粒度の粗粉を得た。この粗粉を同じくジェットミルを用い不活性ガス雰囲気中で微粉砕をして微粉を得た。この微粉は平均粒径 $3.8\mu\text{m}$ (F. S. S. S.) であり、含有酸素量は 4900ppm であった。次に、この微粉を配向磁場強度 15kOe 、成形圧力 1.5ton/cm^2 の条件下で磁場中プレス成形し、 $30\times 20\times 15$ の成形体を作製した。この成形体は実質的に真空の条件で $1080^\circ\text{C}\times 3\text{hr}$ の焼結を行い、得られた焼結体に $900^\circ\text{C}\times 2\text{hr}$ の第1次熱処理、次いで $530^\circ\text{C}\times 2\text{hr}$ の第2次熱処理を施した。得られた焼結体の密度は $7.55\sim 7.58\text{g/cc}$ 、また含有酸素量は 4400ppm であった。これら試料について、常温磁気特性、および平均粒径を測定し、図7に示すような結果を得た。図7に示されるようにNbを含有させることにより焼結時の結晶粒成長を抑制でき、その結果焼結体平均粒径を小さくできる。また、この効果により保磁力 iH_c の向上を期待できる。2.0wt %以上の含有によっても平均粒径の減少をさほど期待出来ず、また最大エネルギー積(BH) maxの低下も大きくなるので0.4~2.0wt %の添加が適量である。

【0020】(実施例8) 金属Nd、金属Dy、Fe、Co、ferro-B、ferro-Nb、金属Gaを所定の重量秤量し、これを真空溶解して重量 10kg のインゴットを作製した。このインゴットの成分分析を行なうと重量比で以下のような組成であった。Nd 27.5-Dy 4.0-B1.04-Nb 0.59-Ga 0.19-Co a-Al b-Fe bal.

① a=0 b=0

材 質	密着強度 (Kg f/cm^2)
① (Co無添加)	180
② (Co添加)	680
③ (Co, Al添加)	700

*果を得た。図6に示されるようにGaを含有させることによって保磁力 iH_c 及び H_k の向上が認められるので0.05wt %以上の含有が必須となる。しかしながら、Gaが0.4wt %を超えると H_k が低下しヒステリシスループの角形性が低下するので上限は1.0wt %ではあるが、好ましくは0.8wt %、より好ましくは0.6wt %、更に好ましくは0.4wt %である。

【0019】(実施例7) ジジムメタル (Nd 70wt % - Pr 30wt %)、金属Dy、Fe、Co、ferro-B、ferro-Nb、金属Gaを所定の重量秤量し、これを真空溶解して重量各 10kg のインゴットを作製した。このインゴットの成分分析を行なうと重量比で以下のような組成であった。

② a=2.02 b=0

③ a=2.10 b=0.34 (wt %)

各々のインゴットをハンマーで解砕した後、さらに粗粉砕機を用い不活性ガス雰囲気中で粗粉砕を行い $500\mu\text{m}$ 以下の粒度の粗粉を得た。この粗粉を同じくジェットミルを用い不活性ガス雰囲気中で微粉砕をして微粉を得た。この微粉は平均粒径 $3.8\mu\text{m}$ (F. S. S. S.) であり、含有酸素量は $6000\sim 6400\text{ppm}$ であった。次に、この微粉を配向磁場強度 15kOe 、成形圧力 1.5ton/cm^2 の条件下で磁場中プレス成形し、 $30\times 20\times 15$ の成形体を作製した。この成形体は実質的に真空の条件で $1100^\circ\text{C}\times 2\text{hr}$ の焼結を行い、得られた焼結体に $900^\circ\text{C}\times 2\text{hr}$ の第1次熱処理、次いで $500\sim 600^\circ\text{C}\times 2\text{hr}$ の第2次熱処理を施した。得られた焼結体の密度は $7.56\sim 7.59\text{g/cc}$ 、また含有酸素量は $5400\sim 5900\text{ppm}$ であった。これら試料について常温磁気特性を測定し、図8に示されるような結果を得た。図8に示されるように、Coを単独で添加したものはCo及びAl無添加のものと比較して第2次熱処理温度依存性が大きくなる。これでは、工業生産上安定した特性の製品をつくるのが困難である。そこで、Co及びAlを複合添加すると図のように第2次熱処理温度依存性を小さくすることができ、この問題を回避することができる。次に前記① (Co無添加)、② (Co添加)、③ (Co, Al添加)の組成を有する磁石にNiメッキを施して、その密着性を評価した。Niメッキは、ワット浴による電解メッキで膜厚 $10\mu\text{m}$ とした。メッキ処理後水洗いして 100°C で5分間乾燥後メッキ密着性試験を行った。結果は下記の通りであり、Co添加材が優れたメッキ密着性を有する。

【0021】

【発明の効果】以上、実施例に示したようにNd-Fe-Co-B型焼結磁石にGa、Al、Nbを複合添加し、Dy量を適正範囲で添加することにより、高保磁力かつ高エネルギー積を有し、高耐熱性、高耐食性を有する磁石を安定的に得ることができる。

【図面の簡単な説明】

【図1】Nd-Fe-Co-B型焼結磁石の全希土類量に対する最大エネルギー積 $(BH)_{max}$ 、腐食減量の変化を示したグラフ。

【図2】Nd-Fe-Co-B型焼結磁石の含有酸素量に対する最大エネルギー積 $(BH)_{max}$ 、保磁力 iH_c の変化を示したグラフ。

【図3】Nd-Fe-Co-B型焼結磁石のGa含有量(0~1.2wt%)に対する最大エネルギー積 $(BH)_{max}$ 、保磁力 iH_c の変化を示したグラフ。

【図4】Nd-Fe-Co-B型焼結磁石のGa含有量

に対する最大エネルギー積 $(BH)_{max}$ 、保磁力 iH_c 、腐食減量の変化を示したグラフ。

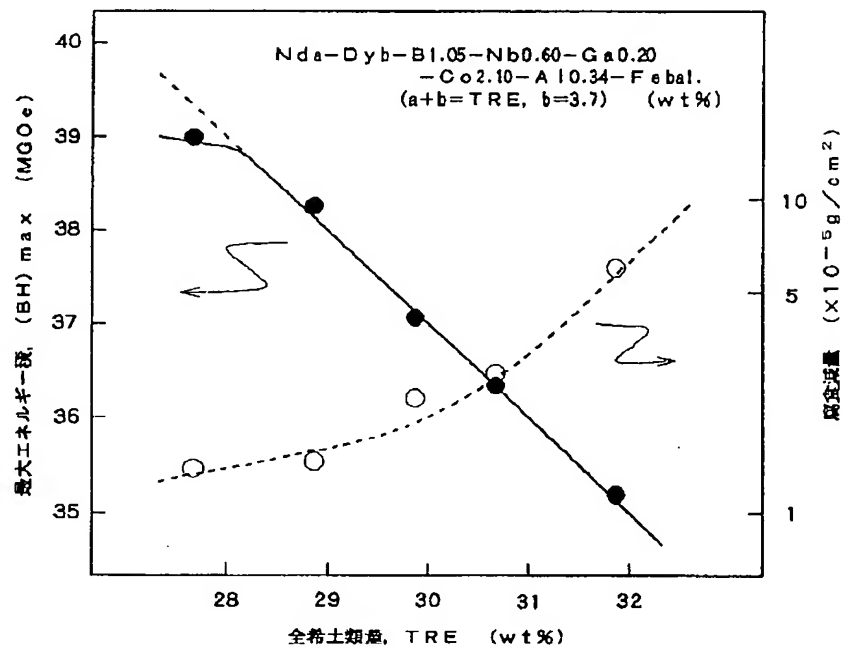
【図5】Nd-Fe-Co-B型焼結磁石のGa含有量(0~0.6wt%)及びDy含有量に対する最大エネルギー積 $(BH)_{max}$ 、保磁力 iH_c の変化を示したグラフ。

【図6】Nd-Fe-Co-B型焼結磁石のGa含有量(0~0.6wt%)に対する最大エネルギー積 $(BH)_{max}$ 、保磁力 iH_c 、角型性の変化を示したグラフ。

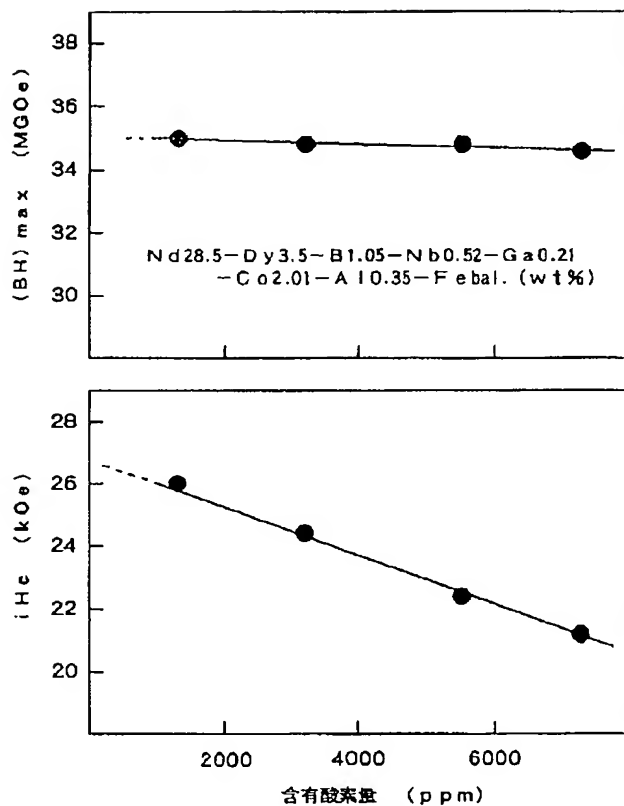
【図7】Nd-Fe-Co-B型焼結磁石のNb含有量に対する焼結体平均結晶粒径、最大エネルギー積 $(BH)_{max}$ の変化を示したグラフ。

【図8】Nd-Fe-Co-B型焼結磁石のCo、Al添加による第2次熱処理温度依存性の変化を示したグラフ。

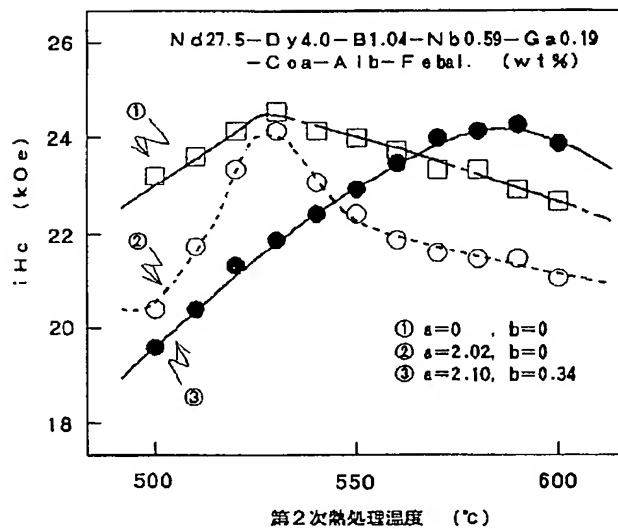
【図1】



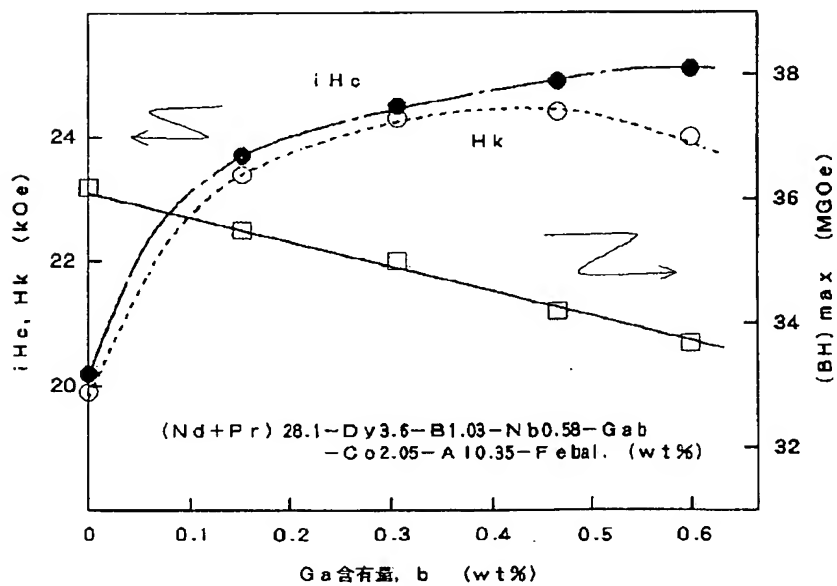
【図2】



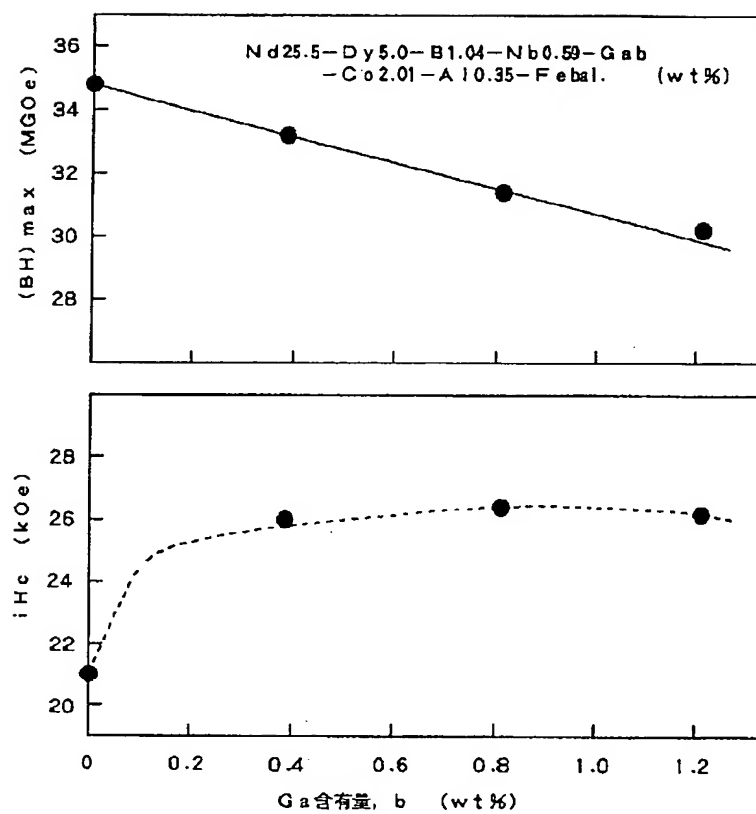
【図8】



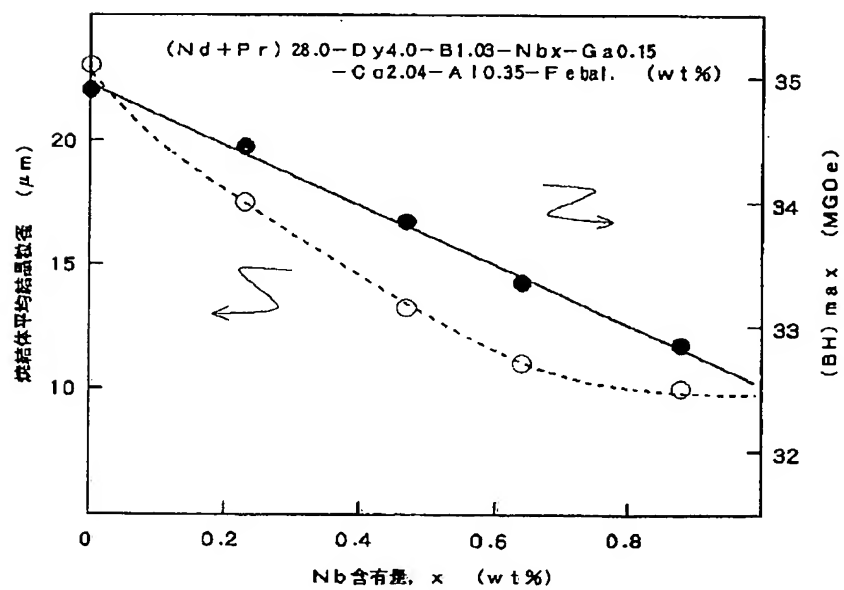
【図6】



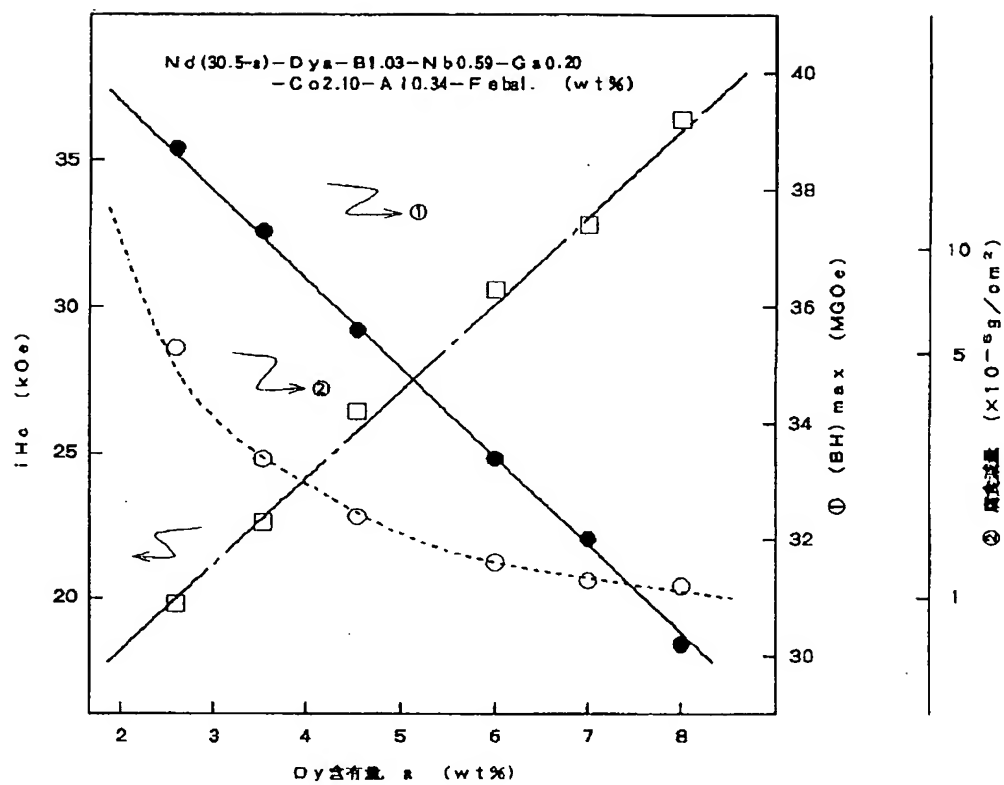
【図3】



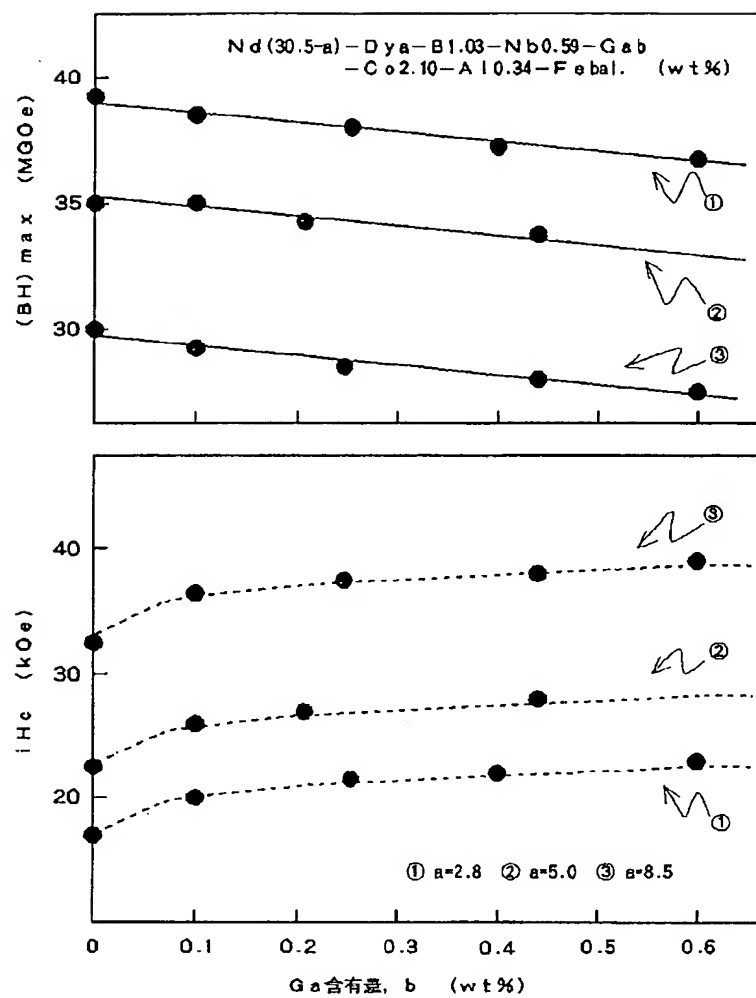
【図7】



【図4】



【図5】



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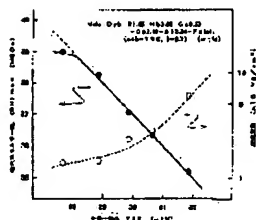
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(21)Application number : 04-249113 (71)Applicant : HITACHI METALS LTD

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(54) ND-FE-CO-B TYPE SINTERED MAGNET



(57)Abstract:

PURPOSE: To obtain a Nd-Fe-Co-B type sintered magnet excellent in corrosion resistance and heat resistance.

CONSTITUTION: A Nd-Fe-Co-B type sintered magnet which comprises 28 to 32wt.% of R (where R is at least one selected from Y and rare earth elements,

and 3.0 to 8.0wt.% of R is Dy and the remainder of R is one or two of Nd and Pr, Nd being contained in R by 50 at% or more), 0.1 to 1.0wt.% of Al, 0.5 to 2.0wt.% of B, 0.1 to 2.0wt.% of Nb, and unavoidable impurities, with the remainder being composed of a composition mainly consisting of Fe. In addition, having a coercive force iH_c of 20kOe or greater and a maximum magnetic energy product (BH) max of 30MGOe or greater, it is excellent in corrosion resistance and heat resistance.

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CLAIMS

[Claim(s)]

[Claim 1] 28 - 32wt% R (however, R is at least one sort chosen from Y and rare earth elements 3.0 - 8.0wt% among R Dy and the remainder) Are one sort of Nd or Pr, or two sorts, and more than 50at% is included for Nd in the inside of R. Co not more than 5.0wt% (however, Co is surely included), 0.1 - 1.0wt% aluminum, A 0.5 - 2.0wt% B, a 0.1 - 2.0wt% Nb, 0.05 - 1.0wt% Ga, The Nd-Fe-Co-B mold sintered magnet which contained 1000 ppm - 6000 ppm oxygen and an unescapable impurity, and the remainder mainly consisted of Fe, and was excellent in the corrosion resistance whose coercive force iH_c is 20 or more kOes, and whose maximum magnetic energy (product BH) max is 30 or more MGOes, and thermal resistance.

[Claim 2] The Nd-Fe-Co-B mold sintered magnet according to claim 1 whose Ga content is 0.05 - 0.8wt%.

[Claim 3] The Nd-Fe-Co-B mold sintered magnet according to claim 1 whose Ga content is 0.1 - 0.6wt%.

[Claim 4] The Nd-Fe-Co-B mold sintered magnet according to claim 1 whose Ga content is 0.1 - 0.4wt%.

[Claim 5] The Nd-Fe-Co-B mold sintered magnet according to claim 1 to 4 whose 3.0 - 5.0wt% is Dy among R and whose maximum magnetic energy (product BH) max is 35 or more MGOes.

[Claim 6] The Nd-Fe-Co-B mold sintered magnet according to claim 1 to 4 whose

5.0 - 8.0wt% is Dy among R and whose coercive force H_c is 25 or more kOes.
[Claim 7] as ordinary temperature magnetic properties -- $H_c \geq 20$ kOe and $(BH)_{max} \geq 30$ MGOe -- it is -- the residual magnetic flux density B_r of 23 to 120 degrees C, and coercive force H_c -- the Nd-Fe-Co-B mold sintered magnet according to claim 1 each temperature coefficients α and β of whose are -
 $0.12 \leq \alpha \leq -0.08\%/^{\circ}\text{C}$ - $0.65 \leq \beta \leq -0.40\%/^{\circ}\text{C}$.
[Claim 8] The Nd-Fe-Co-B mold sintered magnet according to claim 1 to 7 which performed nickel plating to the front face.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention relates to the Nd-Fe-Co-B mold sintering permanent magnet which has the especially excellent corrosion resistance and thermal resistance about the permanent magnet which uses neodymium (Nd), iron (Fe), cobalt (Co), and boron (B) as a principal component.

[0002]

[Description of the Prior Art] Since a Nd-Fe-B mold sintered magnet and a Nd-

Fe-Co-B mold sintered magnet have high energy product (BH) max as compared with a SmCo5 mold sintered magnet or an Sm2Co17 mold sintered magnet, they are used for various applications. However, since a Nd-Fe-B mold sintered magnet and a Nd-Fe-Co-B mold sintered magnet are inferior to thermal stability as compared with these Sm-Co mold sintered magnet, and the thermal stability is increased, various attempts are proposed. In JP,64-7503,A, as a good permanent magnet of thermal stability, general formula: $R(Fe_{1-x-y-z}Co_xByGaz) A$ (However, R is at least one sort chosen from rare earth elements, and is $0 \leq x \leq 0.7$, $0.02 \leq y \leq 0.3$, $0.001 \leq z \leq 0.15$, and $4.0 \leq A \leq 7.5$.) And $R(Fe_{1-x-y-z}Co_xByGazMu) A$ (However, R is at least one sort chosen from rare earth elements, and M is one sort or two sorts or more of elements chosen from Nb, W, V, Ta, and Mo, and is $0 \leq x \leq 0.7$, $0.02 \leq y \leq 0.3$, $0.001 \leq z \leq 0.15$, $u \leq 0.1$, and $4.0 \leq A \leq 7.5$.) What is expressed is indicated.

[0003]

[Problem(s) to be Solved by the Invention] However, when it was going to produce stably the Nd-Fe-Co-B mold anisotropy sintered magnet which has high corrosion resistance and a high heatproof property, and has the coercive force iH_c of a high level, and energy product (BH) max in coincidence, it turned out exceeding said well-known technique that it limits [the limitation of the component presentation range based on still more detailed research and examination, / of an oxide]. Especially this invention offers the Nd-Fe-Co-B mold sintered magnet excellent in corrosion resistance and thermal resistance based on such knowledge. When this invention uses Co, Dy, and Nb effectively, coercive force iH_c offers stably the large Nd-Fe-Co-B mold sintered magnet of energy product (BH) max greatly by raising corrosion resistance remarkably, giving high thermal resistance by making the amount of D(ies), and the amount of Ga(s) into the specific range, and lowering the amount of rare earth R to coincidence, and limiting the amount of oxygen.

[0004]

[Means for Solving the Problem] This invention is 28 - 32wt% R (however, R is at

least one sort chosen from Y and rare earth elements 3.0 - 8.0wt% among R Dy and the remainder). Are one sort of Nd or Pr, or two sorts, and more than 50at% is included for Nd in the inside of R. A 5.0wt(s)% following Co (however, Co is surely included), 0.1 - 1.0wt% aluminum, A 0.5 - 2.0wt% B, a 0.1 - 2.0wt% Nb, 0.05 - 1.0wt% Ga, It is the Nd-Fe-Co-B mold sintered magnet which contained 1000 ppm - 6000 ppm oxygen and an unescapable impurity, and the remainder mainly consisted of Fe, and was excellent in the corrosion resistance whose coercive force iH_c is 20 or more kOes, and whose maximum magnetic energy (product BH) max is 30 or more MGOes, and thermal resistance. The reason for limitation of a presentation of the permanent magnet of this invention is explained to a detail below.

[0005] In this invention, R is contained in 28 - 32wt%. It is so effective in (BH) max and corrosion resistance improvement that there are few amounts of R as less than [32wt%] as shown in the below-mentioned example 4. However, less than [28wt%], it becomes easy to generate alpha-Fe in an ingot, and is hard to expect increase of (BH) max. Therefore, the amount of R is made into 28 - 32wt%. R shall contain Nd beyond 50at% among R components, in order to make Nd into a subject. Although 3.0 - 8.0wt% Dy is contained in the inside of R, the remainder is mixing with a Nd independent, or Nd and Pr. Pr has effectiveness in improvement in coercive force iH_c .

[0006] By making Dy contain as an R component, while Curie point T_c goes up, an anisotropy magnetic field (H_A) increases, coercive force iH_c improves, and thermal resistance is raised remarkably. Moreover, Dy has effectiveness also in corrosion-resistant improvement. In this invention, if there are few contents of Dy than 3.0wt(s)%, the purpose of this invention of raising thermal stability and corrosion resistance will not be attained. However, when a content increases more than 8.0wt%, degradation of a residual magnetic flux density B_r and the magnetic properties by the fall of maximum energy product (BH) max is remarkable. Therefore, the content of Dy is made into 3.0 - 8.0wt%. If a content has more Dy than 5.0wt(s)%, the fall of a residual magnetic flux density B_r and

maximum energy product (BH) max can acquire the coercive force H_c of 25 or more kOes of a certain thing. Therefore, the content of Dy is made into 5.0 - 8.0wt% when it is going to acquire a high coercive force property more.

Conversely, what is necessary is just to make the content of Dy into 3.0 - 5.0wt%, when it is going to obtain a big residual magnetic flux density B_r and big maximum energy product (BH) max.

[0007] While improving own corrosion resistance of a magnet alloy, without Co almost reducing a residual magnetic flux density B_r in this invention, it is effective in raising corrosion resistance by improving the adhesion of nickel plating which is corrosion protection coating. Moreover, Fe in the main phase ($Nd_2Fe_{14}B$) It is effective in raising curie point T_c by permuting by Co. However, if the amount of permutations of Co is made [many], the big and rough crystal grain which considers abnormality grain growth at the time of sintering as a cause will be generated, and coercive force H_c and the square shape nature of a hysteresis curve will fall. Therefore, Co content is made into less than [5.0wt%].

[0008] In this invention, aluminum has the effectiveness which eases the temperature conditions at the time of heat treatment of Co add-in material. That is, when Co is contained, the fluctuation of magnetic properties or thermal stability of an ingredient is large to fluctuation of heat treatment temperature. Even if it changes heat treatment conditions somewhat, magnetic properties and thermal stability stop changing, if aluminum of optimum dose is added there. The production control of a permanent magnet becomes easy by this, and the permanent magnet by which quality was stabilized can be efficiently produced now. As for the above-mentioned effectiveness, less than [0.1wt%] is [the content of aluminum] inadequate. On the other hand, if 1.0wt% is exceeded, the fall of a residual magnetic flux density B_r will become remarkable. Therefore, the content of aluminum is made into 0.1 - 1.0wt%. If B exceeds 2.0wt% on the other hand by not acquiring high coercive force in the case of below 0.5wt%, the nonmagnetic phase which is rich in B will increase, and a residual magnetic flux density B_r will fall. Therefore, it may be 0.5 - 2.0wt%. The content of desirable B

is 0.8 - 1.2wt%.

[0009] Ga does not almost reduce a residual magnetic flux density B_r , and is effective in improving coercive force iH_c . When Ga content is less than [0.05wt%], the effectiveness of improving coercive force iH_c is not enough. If Ga content exceeds 1.0wt(s)%, a residual magnetic flux density B_r will fall and a desired high energy product will not be obtained. Therefore, Ga content is made into 0.05 - 1.0wt%. Since the square shape nature of a magnetic hysteresis curve will worsen if there are many Ga contents, also in order to give high square shape nature, the desirable content of Ga is 0.05 - 0.8wt%. The more desirable content of Ga is 0.1 - 0.6wt%. Furthermore, it is 0.1 - 0.4wt% preferably.

[0010] The permanent magnet of this invention contains 0.1 - 2.0wt% Nb other than the above-mentioned component. Nb has the effectiveness which controls that crystal grain makes it big and rough at the time of sintering. According to this effectiveness, coercive force iH_c improves and the square shape nature of a hysteresis curve becomes good. Moreover, that the crystal grain of a sintered compact becomes detailed contributes to magnetic good magnetization nature greatly, and the Nd-Fe-Co-B mold sintered magnet with still better magnetization nature has the outstanding thermal resistance. Therefore, Nb is an effective additive at the magnet which has thermal resistance. When the content of Nb is less than [0.1wt%], the effectiveness which controls a big and rough grain is inadequate. On the other hand, when the content of Nb exceeds 2.0wt(s)%, many nonmagnetic borides of Nb or Nb-Fe occur, and it falls [a residual magnetic flux density B_r and Curie point T_c] remarkably and is not desirable. Therefore, the content of Nb is made into 0.1 - 2.0wt%. Preferably, it is 0.1 - 1.0wt%.

[0011] An oxygen content is set to 1000 ppm - 6000 ppm. When there is less oxygen than 1000 ppm, there is industrial production top risk that magnet powder and its consolidation object tend to ignite. On the other hand, in [than 6000 ppm] more, oxygen reacts with a rare earth R component, a rare earth oxide is formed, and it becomes difficult to obtain the magnet of high coercive force and a

high energy product.

[0012] The sintered magnet of this invention can be manufactured as follows.

That is, the ingot which has a fixed component presentation is manufactured by vacuum melting, and coarse powder with a particle size of about 500 micrometers is obtained by next carrying out coarse grinding of this ingot. This coarse powder is pulverized in an inert gas ambient atmosphere using a jet mill, and fines with a mean particle diameter of 3.0-6.0 micrometers (F. S.S.S.) are obtained. Next, it is after press forming in a magnetic field, and a 1000-1150-degree C temperature requirement about these fines under the conditions of orientation magnetic field 15kOe and compacting pressure 1.5 ton/cm², and is

*****. **. Heat treatment after sintering can be performed as follows. The sintered compact which sintered and acquired the Plastic solid is once cooled to a room temperature. The cooling rate after sintering hardly affects the coercive force iH_c of a final product. Subsequently, it heats in temperature of 800-1000 degrees C, and holds for 0.2 to 5 hours. This is considered as the first heat treatment. When whenever [stoving temperature] exceeds less than 800 degrees C or 1000 degrees C, sufficient high coercive force is not acquired. It cools to a room temperature thru/or the temperature of 600 degrees C after heating maintenance with 0.3-50-degree-C cooling rate for /. When a cooling rate exceeds a part for 50-degree-C/, a balanced phase required for aging is not obtained and sufficient high coercive force is not acquired. Moreover, by 0.3-degree-C/, heat treatment takes time amount to the cooling rate of the following, and it is not economical on industrial production. Preferably, 0.6-2.0-degree-C cooling rate for /is chosen. Although cooling termination temperature has a desirable room temperature, as long as it sacrifices coercive force iH_c for some, it may carry out to to 600 degrees C, and below the temperature may quench. Preferably, it cools to ordinary temperature -400 degree C temperature. Heat treatment is performed at the temperature of further 500-650 degrees C for 0.2 to 3 hours. This is considered as the second heat treatment. Although it changes with presentations, heat treatment at 540-640 degrees C is preferably effective.

when heat treatment temperature is less than 500 degrees C, and when higher than 650 degrees C, even if high coercive force is acquired, decline in an irreversible demagnetizing factor cuts. After heat treatment is cooled like the first heat treatment with 0.3-400-degree-C cooling rate for /. Cooling can be performed by the argon air-current middle class underwater and among a silicone oil. When a cooling rate exceeds a part for 400-degree-C/, a crack goes into a sample by quenching and the permanent magnet ingredient which is industrially worthy is not obtained. Moreover, in the following, the phase which is not desirable appears in coercive force iH_c in a cooling process by 0.3-degree-C/. [0013]

[Example] Hereafter, an example explains this invention to a detail further.

(Example 1) Predetermined carried out weight weighing capacity of Metal Nd, Metals Dy, Fe, and Co, ferro-B, ferro-Nb, and the metal Ga, vacuum melting of this was carried out, and the ingot with a weight of 10kg was produced. When component analysis of this ingot was performed, they were the following presentations in the weight ratio. Nd27.5-Dy3.6-B1.03-Nb0.58-Ga0.18-Co2.02-Al0.35-Fe bal. (wt%)

After cracking this ingot with a hammer, coarse grinding in the inside of an inert gas ambient atmosphere was further performed using the coarse crusher, and coarse powder with a grain size of 500 micrometers or less was obtained.

Similarly this coarse powder was pulverized in the inert gas ambient atmosphere using the jet mill, and fines were obtained. These fines were 4.0 micrometers (F. S.S.S.) in mean particle diameter, and the amount of content oxygen was 5500 ppm. Next, press forming in a magnetic field of these fines was carried out under the conditions of orientation magnetic-field-strength 15kOe and compacting pressure 1.5 ton/cm², and the Plastic solid of 30x20x15 was produced. This Plastic solid sintered 1080 degree-Cx3hr on condition that the vacuum substantially, was first-heat-treated [of 900 degree-Cx2hr] to the obtained sintered compact, ranked second to it, and performed the second heat treatment of 530 degree-Cx2hr. 7.55g [cc] /and the amount of content oxygen of the

consistency of the obtained sintered compact were 4800 ppm. The following values were acquired when the ordinary temperature magnetic properties of this sample were measured.

$B_r = 12.6 \text{ kG}$, $B_H = 11.6 \text{ kOe}$, $iH_c = 21.8 \text{ kOe}$, $(BH)_{\max} = 31.3 \text{ MGOe}$ and $\alpha = -0.10\%/\text{degree C}$ value were further acquired as Curie point $T_c = 35.6 \text{ MGOe}$ as temperature coefficients α and β of B_r and iH_c of 23 to 120 degrees C. [340 degrees C and] Moreover, the 100-degree C irreversible demagnetizing factor of the sample which carried out permeance coefficient $P_c = 1.0$ and 2.0 configuration has the thermal resistance which is 2.1 and 1.1% respectively, and was excellent.

[0014] (Example 2) Experiment conditions were changed and the following experimental result was obtained like the example 1.

Presentation : Nd25.5-Dy6.4-B1.04-Nb0.55-Ga0.22-Co2.00-aluminum0.36-Febal. (wt%)

sintering : The 1100 degree-Cx2hr first heat treatment : The 900 degree-Cx2hr second heat treatment : 530-degree-Cx2hr ordinary temperature magnetic properties : $B_r = 11.4 \text{ kG}$, $B_H = 11.0 \text{ kOe}$, $iH_c = 27.8 \text{ kOe}$, $(BH)_{\max} = 31.3 \text{ MGOe}$ Curie point : $T_c = 340\text{-degree-C}$ irreversible demagnetizing factor [at 100 degree C] : $P_c = 1.0 \rightarrow 1.8\%$, $P_c = 2.0 \rightarrow 0.8\%$ B_r temperature coefficient (α), iH_c temperature coefficient (β) [23 degrees C - 120 degrees C] : $\alpha = -0.09\%/\text{degree-C}$, $\beta = -0.51\%/\text{degree C}$ sintered compact content oxygen : Like the 5800 ppm example 1, it excels in the elevated-temperature property with ordinary temperature magnetic properties, and the magnet excellent in thermal resistance can be obtained.

[0015] (Example 3) Didym metal (Nd70wt%-Pr30wt%) was used and the following experimental result was obtained like examples 1 and 2.

Presentation : Nd18.9-Pr5.1-Dy7.3-B1.10-Nb0.71-Ga0.37-Co4.72-aluminum0.33-Febal. (wt%)

sintering : The 1080 degree-Cx2hr first heat treatment : The 900 degree-Cx2hr second heat treatment : 520-degree-Cx2hr ordinary temperature magnetic

properties : $Br = 11.5 \text{ kG}$, $H_c = 10.9 \text{ kOe}$, $iH_c = 30.0 \text{ kOe}$, $(BH)_{\text{max}} = 31.2 \text{ MGOe}$
 Curie point : $T_c = 375\text{-degree-C}$ irreversible demagnetizing factor [at 100 degree C] : $P_c = 1.0 \rightarrow 1.4\%$, $P_c = 2.0 \rightarrow 0.5\%$, Br temperature coefficient (α), iH_c temperature coefficient (β) [$23\text{ degrees C} - 120\text{ degrees C}$] : $\alpha = -0.09\%/\text{degree-C}$, $\beta = -0.48\%/\text{degree C}$ sintered compact content oxygen : Even when 5400 ppm didym metal is used The magnet which was excellent in ordinary temperature magnetic properties, an elevated-temperature property, and thermal resistance like examples 1 and 2 can be obtained.

[0016] (Example 4) Predetermined carried out weight weighing capacity of Metal Nd, Metals Dy, Fe, and Co, ferro-B, ferro-Nb, and the metal Ga, vacuum melting of this was carried out, and the ingot with a weight of 10kg each was produced. When component analysis of this ingot was performed, they were the following presentations in the weight ratio.

Nd_a-Dy_b-B_{1.05}-Nb_{0.58}-Ga_{0.20}-Co_{0.20}-Al_{0.33}-Fe_{bal.} ($a+b=1$, $b=3.7$) (wt%)

After cracking each ingot with a hammer, coarse grinding in the inside of an inert gas ambient atmosphere was further performed using the coarse crusher, and coarse powder with a grain size of 500 micrometers or less was obtained.

Similarly this coarse powder was pulverized in the inert gas ambient atmosphere using the jet mill, and fines were obtained. These fines were 3.7 micrometers (F. S.S.S.) in mean particle diameter, and the amount of content oxygen was 1500-5000 ppm. Next, press forming in a magnetic field of these fines is carried out under the conditions of orientation magnetic-field-strength 15kOe and compacting pressure 1.5 ton/cm², and it is the Plastic solid of 30x20x15. It produced. This Plastic solid sintered 1070 degree-Cx2hr on condition that the vacuum substantially, was first-heat-treated [of 900 degree-Cx2hr] to the obtained sintered compact, ranked second to it, and performed the second heat treatment of 540 degree-Cx2hr. 7.55-7.58g [cc] /and the amount of content oxygen of the consistency of the obtained sintered compact were 1000-4000 ppm. The result as measures how maximum energy product $(BH)_{\text{max}}$ and

corrosion weight loss change to a TRE content about this sample and shows it to drawing 1 was obtained. Corrosion weight loss is obtained when a magnet is exposed for 100 hours into the temperature of 120 degrees C, 90% of humidity, and the environment of atmospheric-pressure 1.0atm. As shown in drawing 1 , (BH) max can be improved by lessening the amount of TRE(s), but if it is less than [28wt%], it becomes easy to generate alpha-Fe in an ingot, and is hard to expect increase of (BH) max. It can be made to decrease by lessening the amount of corrosion-weight-loss mist beam TRE(s). This is for the Nd-rich phase which is easy to corrode by lessening TRE to decrease. However, since reduction of coercive force iH_c will become remarkable if the amount of content oxygen exceeds 6000 ppm also as a low value of 28 - 32wt% for the amount of TRE(s), the amount of oxygen is set to 1000-6000 ppm. The oxygen content in a sintered magnet and the relation of magnetic properties are shown in drawing 2 . [0017] (Example 5) Predetermined carried out weight weighing capacity of Metal Nd, Metals Dy, Fe, and Co, ferro-B, ferro-Nb, and the metal Ga, vacuum melting of this was carried out, and the ingot with a weight of 10kg each was produced. When component analysis of this ingot was performed, they were the following presentations in the weight ratio.

$Nd_{30.5-a}Dy_bB_{1.03}Nb_{0.59}Ga_bCo_{2.10}Al_{0.34}Fe_{bal}$. ($2.8 \leq a \leq 8.5$, $0 \leq b \leq 1.2$) (wt%)

After cracking each ingot with a hammer, coarse grinding in the inside of an inert gas ambient atmosphere was further performed using the coarse crusher, and coarse powder with a grain size of 500 micrometers or less was obtained.

Similarly this coarse powder was pulverized in the inert gas ambient atmosphere using the jet mill, and fines were obtained. These fines were 3.8 micrometers (F. S.S.) in mean particle diameter, and the amount of content oxygen was 5500-6400 ppm. Next, press forming in a magnetic field of these fines is carried out under the conditions of orientation magnetic-field-strength 15kOe and compacting pressure 1.5 ton/cm², and it is the Plastic solid of 30x20x15. It produced. This Plastic solid sintered 1100 degree-Cx2hr on condition that the

vacuum substantially, was first-heat-treated [of 900 degree-Cx2hr] to the obtained sintered compact, ranked second to it, and performed the second heat treatment of 580 degree-Cx2hr. 7.55-7.59g [cc] /and the amount of content oxygen of the consistency of the obtained sintered compact were 5000-5900 ppm. About these samples, ordinary temperature magnetic properties were measured and the result as shown in drawing 3 R> 3, drawing 4 , and drawing 5 was obtained. Although drawing 3 is the result of measuring as Dy=5.0wt%, less than [0.05wt%], Ga content cannot demonstrate effectiveness easily, but since the improvement in coercive force iHc is not so much expectable on the other hand only by reduction of maximum energy product (BH) max becoming remarkable as for more than 1.0wt%, 0.05 - 1.0wt% is optimum dose. Since the effectiveness of raising coercive force iHc is large, without Ga falling (BH) max remarkably as compared with Dy, 0.01 - 1.0wt% of content becomes indispensable in this invention. The result to which Dy content was changed, having used Ga content as 0.20wt(s)% at drawing 4 is shown. Although Dy content contributes to improvement in iHc greatly, since it reduces (BH) max remarkably by one side, 3.6 - 8.0wt% of a content is optimum dose. Although the result at the time of changing Ga content to 0 - 0.6wt% by making Dy content into a parameter at drawing 5 is shown, if Dy content exceeds 8.0wt(s)%, (BH) max will fall remarkably. Moreover, it is hard to acquire the high coercive force which exceeds 20kOe as Dy content is less than [3.0wt%].

[0018] (Example 6) Predetermined carried out weight weighing capacity of didym metal (Nd70wt%-Pr30wt%), Metals Dy, Fe, and Co, ferro-B, ferro-Nb, and the metal Ga, vacuum melting of this was carried out, and the ingot with a weight of 10kg each was produced. When component analysis of this ingot was performed, they were the following presentations in the weight ratio.

(Nd+Pr) 28.1-Dy3.6-B1.03-Nb0.58-Gab -Co2.05-aluminum0.35-Febal.

(0<=b<=0.6) (wt%)

After cracking each ingot with a hammer, coarse grinding in the inside of an inert gas ambient atmosphere was further performed using the coarse crusher, and

coarse powder with a grain size of 500 micrometers or less was obtained. Similarly this coarse powder was pulverized in the inert gas ambient atmosphere using the jet mill, and fines were obtained. These fines were 3.7 micrometers (F. S.S.S.) in mean particle diameter, and the amount of content oxygen was 5600 ppm. Next, press forming in a magnetic field of these fines was carried out under the conditions of orientation magnetic-field-strength 15kOe and compacting pressure 1.5 ton/cm², and the Plastic solid of 30x20x15 was produced. This Plastic solid sintered 1080 degree-Cx2.5hr on condition that the vacuum substantially, was first-heat-treated [of 890 degree-Cx2hr] to the obtained sintered compact, ranked second to it, and performed the second heat treatment of 530 degree-Cx2hr. 7.55-7.58g [cc] /and the amount of content oxygen of the consistency of the obtained sintered compact were 2800 ppm. About these samples, ordinary temperature magnetic properties were measured and the result as shown in drawing 6 was obtained. Since improvement in coercive force iHc and Hk is accepted by making Ga contain as shown in drawing 6 , content beyond 0.05wt% becomes indispensable. however -- although an upper limit is 1.0wt(s)% since Hk will fall and the square shape nature of the hysteresis loop will fall, if Ga exceeds 0.4wt(s)% -- desirable -- 0.8wt(s)% -- more -- desirable -- 0.6wt(s)% -- it is 0.4wt(s)% still more preferably.

[0019] (Example 7) Predetermined carried out weight weighing capacity of didym metal (Nd70wt%-Pr30wt%), Metals Dy, Fe, and Co, ferro-B, ferro-Nb, and the metal Ga, vacuum melting of this was carried out, and the ingot with a weight of 10kg each was produced. When component analysis of this ingot was performed, they were the following presentations in the weight ratio.

(Nd+Pr) 28.0-Dy4.0-B1.03-Nbx-Ga0.15 -Co2.04-aluminum0.35-Febal.

(0<=x<=1.0) (wt%)

After cracking each ingot with a hammer, coarse grinding in the inside of an inert gas ambient atmosphere was further performed using the coarse crusher, and coarse powder with a grain size of 500 micrometers or less was obtained.

Similarly this coarse powder was pulverized in the inert gas ambient atmosphere

using the jet mill, and fines were obtained. These fines were 3.8 micrometers (F. S.S.S.) in mean particle diameter, and the amount of content oxygen was 4900 ppm. Next, press forming in a magnetic field of these fines was carried out under the conditions of orientation magnetic-field-strength 15kOe and compacting pressure 1.5 ton/cm², and the Plastic solid of 30x20x15 was produced. This Plastic solid sintered 1080 degree-Cx3hr on condition that the vacuum substantially, was first-heat-treated [of 900 degree-Cx2hr] to the obtained sintered compact, ranked second to it, and performed the second heat treatment of 530 degree-Cx2hr. 7.55-7.58g [cc] /and the amount of content oxygen of the consistency of the obtained sintered compact were 4400 ppm. About these samples, ordinary temperature magnetic properties and mean particle diameter were measured, and the result as shown in drawing 7 R> 7 was obtained. By making Nb contain, as shown in drawing 7 , the grain growth at the time of sintering can be controlled, and, as a result, sintered compact mean particle diameter can be made small. Moreover, improvement in coercive force iH_c is expectable with this effectiveness. 2. Since reduction in mean particle diameter cannot be expected so much by content beyond 0wt% and the fall of maximum energy product (BH) max also becomes large, 0.4 - 2.0wt% of addition is optimum dose.

[0020] (Example 8) Predetermined carried out weight weighing capacity of Metal Nd, Metals Dy, Fe, and Co, ferro-B, ferro-Nb, and the metal Ga, vacuum melting of this was carried out, and the ingot with a weight of 10kg was produced. When component analysis of this ingot was performed, they were the following presentations in the weight ratio. Nd27.5-Dy4.0-B1.04-Nb0.59-Ga0.19-Coa-Alb-Febal.** a=0 b=0 ** a=2.02 b=0 ** a=2.10 b= 0.34 (wt%)

After cracking each ingot with a hammer, coarse grinding in the inside of an inert gas ambient atmosphere was further performed using the coarse crusher, and coarse powder with a grain size of 500 micrometers or less was obtained. Similarly this coarse powder was pulverized in the inert gas ambient atmosphere using the jet mill, and fines were obtained. These fines were 3.8 micrometers (F.

S.S.S.) in mean particle diameter, and the amount of content oxygen was 6000-6400 ppm. Next, press forming in a magnetic field of these fines is carried out under the conditions of orientation magnetic-field-strength 15kOe and compacting pressure 1.5 ton/cm², and it is the Plastic solid of 30x20x15. It produced. This Plastic solid sintered 1100 degree-Cx2hr on condition that the vacuum substantially, was first-heat-treated [of 900 degree-Cx2hr] to the obtained sintered compact, ranked second to it, and performed the second heat treatment of 500-600 degree-Cx2hr. 7.56-7.59g [cc] /and the amount of content oxygen of the consistency of the obtained sintered compact were 5400-5900 ppm. Ordinary temperature magnetic properties were measured about these samples, and the result as shown in drawing 8 was obtained. As shown in drawing 8 , as compared with a thing Co and aluminum additive-free in what added Co independently, the second heat-treatment-temperature dependency becomes large. It is difficult to build the product of the property stabilized on industrial production with this. Then, if compound addition of Co and the aluminum is carried out, as shown in drawing, the second heat-treatment-temperature dependency can be made small, and this problem can be avoided. Next, nickel plating was performed to the magnet which has the presentation of the aforementioned ** (Co additive-free), ** (Co addition), and ** (Co, aluminum addition), and the adhesion was evaluated. nickel plating was made into 10 micrometers of thickness by the electrolytic plating by the Watts bath. It washed in cold water after plating processing and the plating adhesion test after desiccation was performed for 5 minutes at 100 degrees C. The result is as follows and has the plating adhesion excellent in Co add-in material.

** Quality Adhesion reinforcement (Kgf/cm²)

** Co additive-free 180 ** (Co addition) 680 ** (Co, aluminum addition) 700
[0021]

[Effect of the Invention] As mentioned above, the magnet which has high coercive force and a high energy product, and has high thermal resistance and high corrosion resistance can be stably obtained by carrying out compound

addition of Ga, aluminum, and the Nb at a Nd-Fe-Co-B mold sintered magnet, as shown in the example, and adding the amount of D(ies) in the proper range.

[Translation done.]

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1. This document has been translated by computer. So the translation may not reflect the original precisely.
2. **** shows the word which can not be translated.
3. In the drawings, any words are not translated.

DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]

[Drawing 1] Maximum energy product (BH) max to the total amount of rare earth of a Nd-Fe-Co-B mold sintered magnet, the graph which showed change of corrosion weight loss.

[Drawing 2] Maximum energy product (BH) max to the amount of content oxygen of a Nd-Fe-Co-B mold sintered magnet, the graph which showed change of coercive force iH_c .

[Drawing 3] Maximum energy product (BH) max to Ga content (0 - 1.2wt%) of a Nd-Fe-Co-B mold sintered magnet, the graph which showed change of coercive force iH_c .

[Drawing 4] The graph which showed change of maximum energy product (BH) max to Ga content of a Nd-Fe-Co-B mold sintered magnet, coercive force iH_c , and corrosion weight loss.

[Drawing 5] Maximum energy product (BH) max to Ga content (0 - 0.6wt%) and

Dy content of a Nd-Fe-Co-B mold sintered magnet to carry out, the graph which showed change of coercive force iH_c .

[Drawing 6] The graph which showed change of maximum energy product $(BH)_{max}$ to Ga content (0 - 0.6wt%) of a Nd-Fe-Co-B mold sintered magnet, coercive force iH_c , and square shape nature.

[Drawing 7] The diameter of sintered compact average crystal grain to Nb content of a Nd-Fe-Co-B mold sintered magnet, the graph which showed change of maximum energy product $(BH)_{max}$.

[Drawing 8] The graph which showed Co of a Nd-Fe-Co-B mold sintered magnet, and change of the second heat-treatment-temperature dependency by aluminum addition.

[Translation done.]

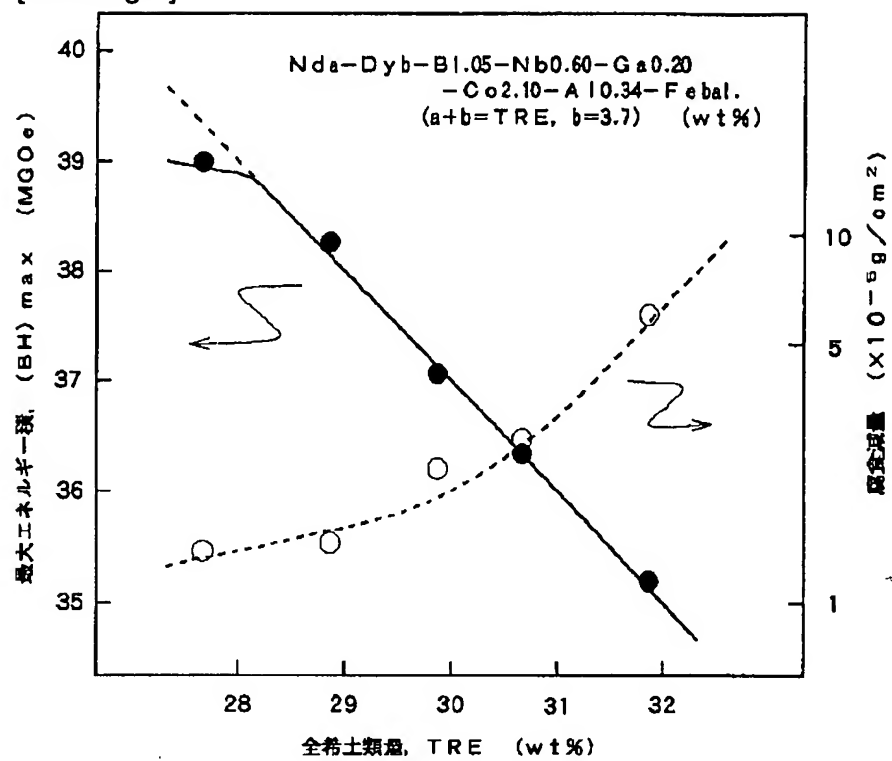
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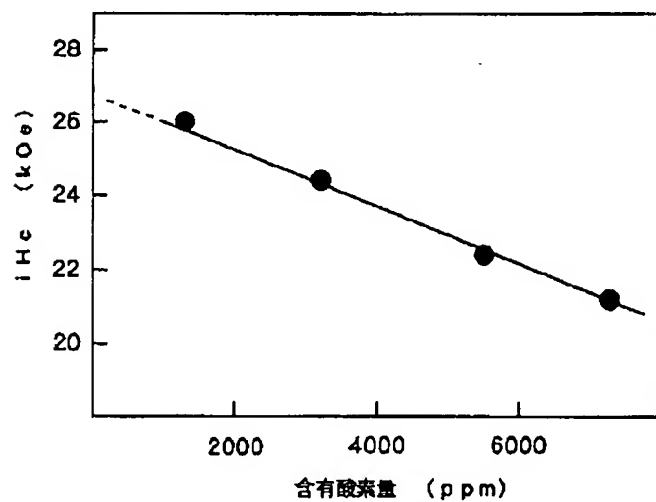
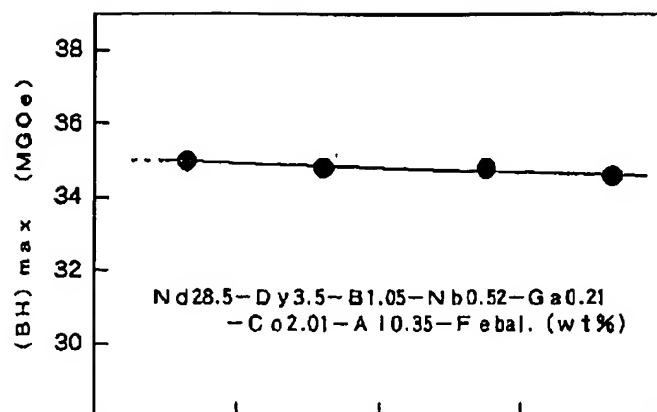
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DRAWINGS

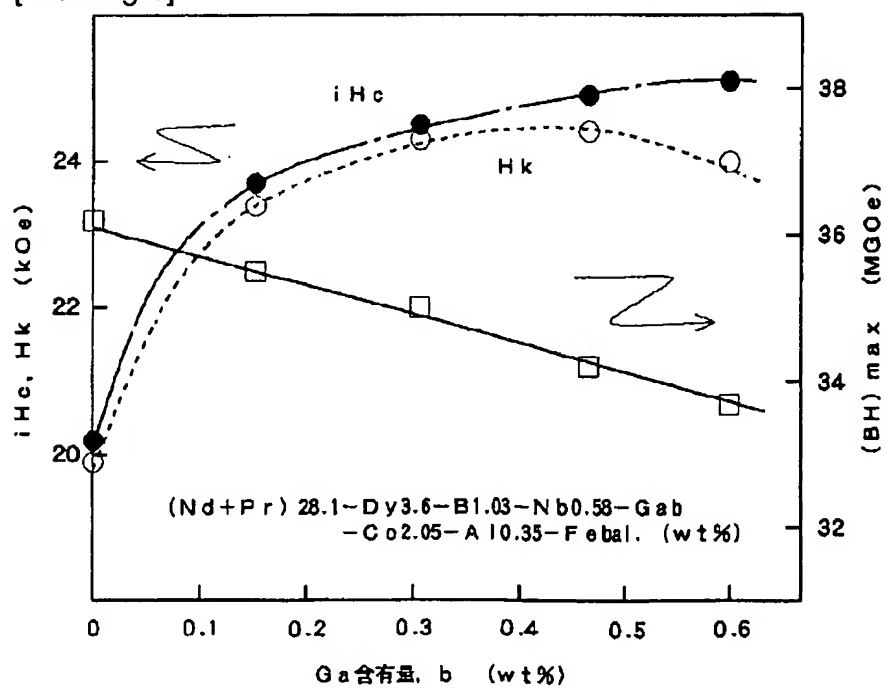
[Drawing 1]



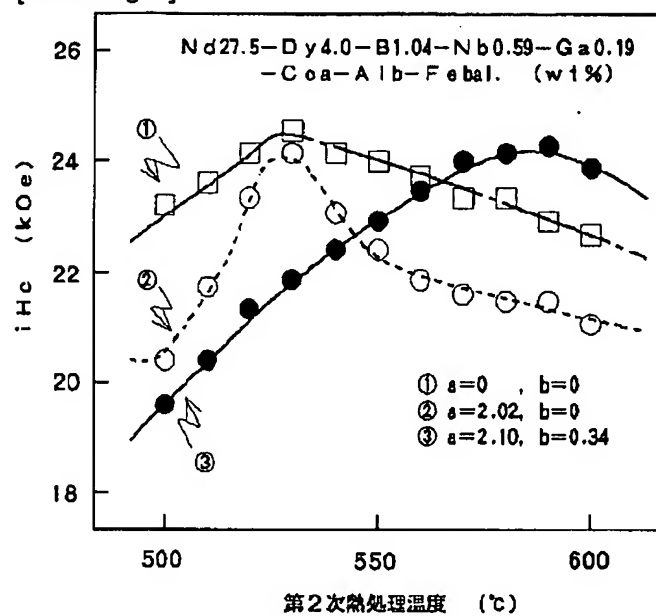
[Drawing 2]



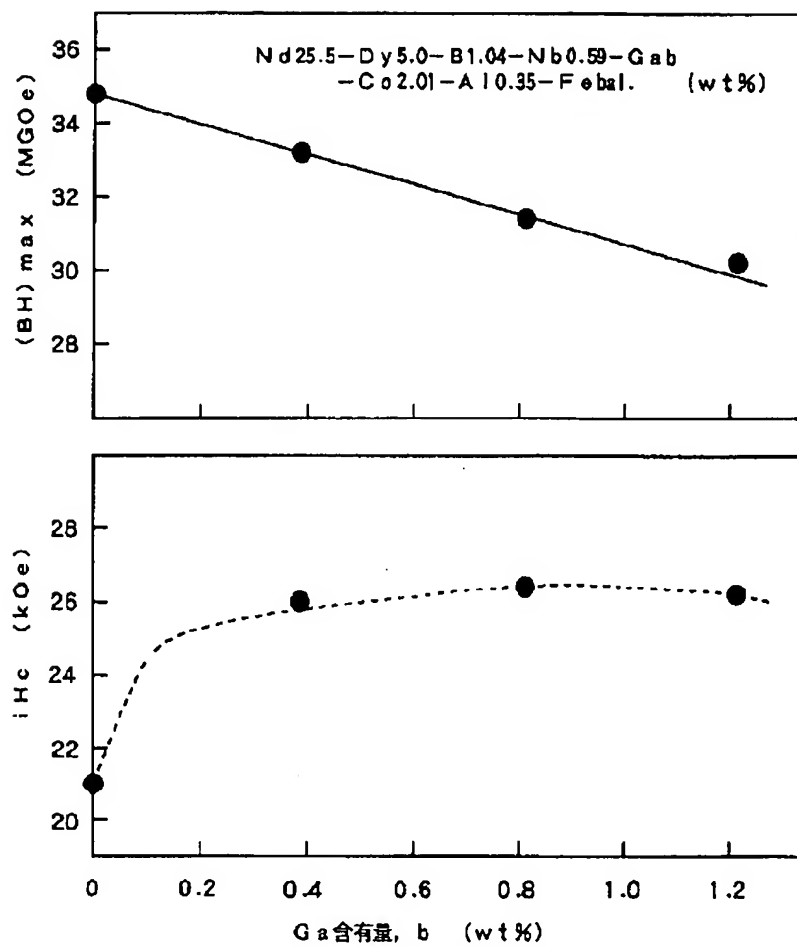
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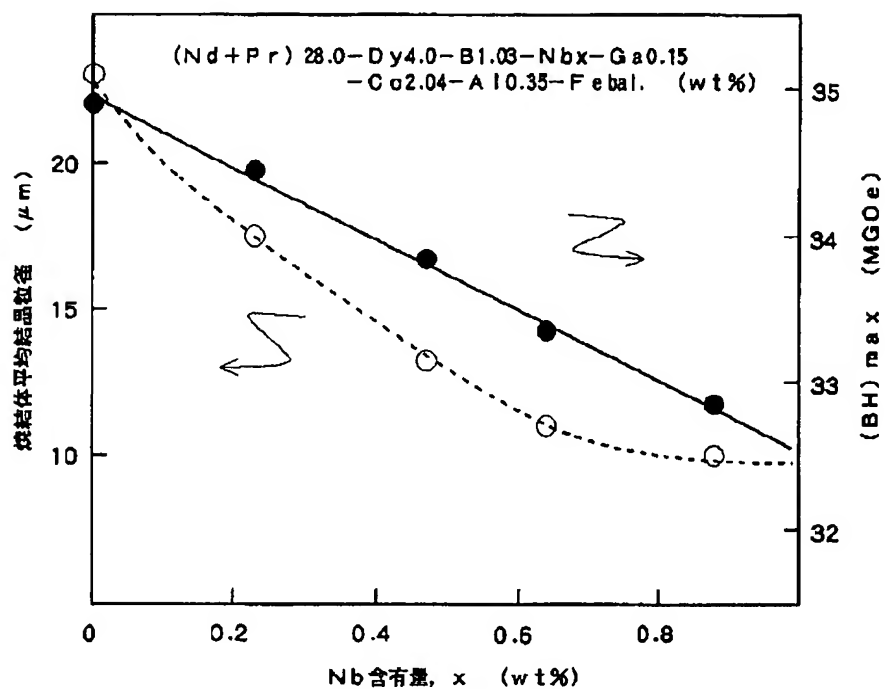
[Drawing 8]



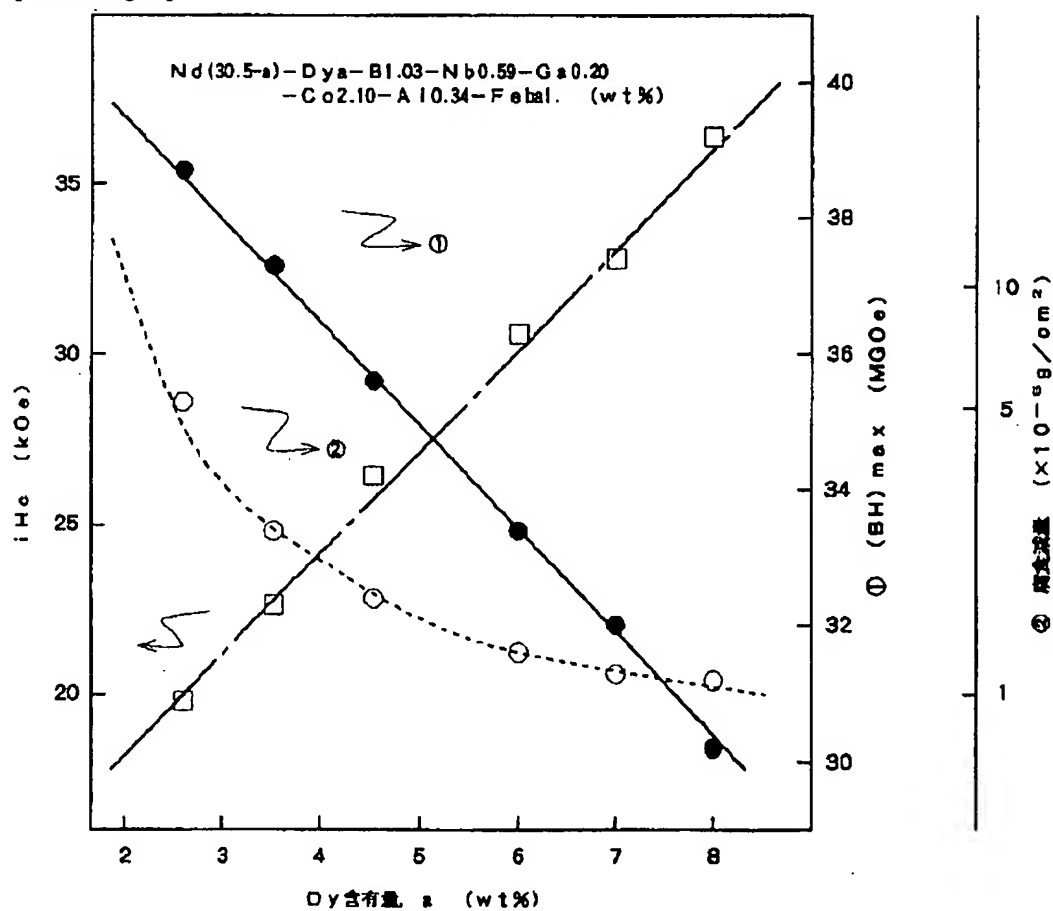
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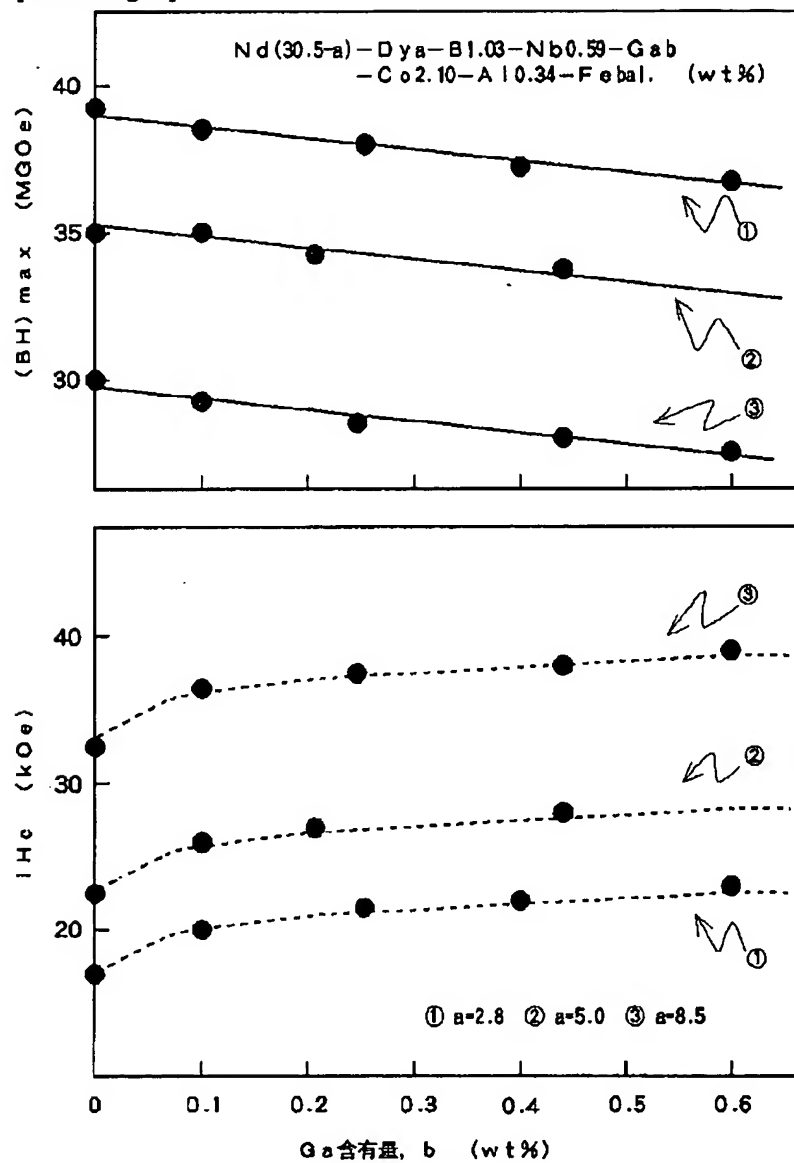
[Drawing 7]



[Drawing 4]



[Drawing 5]



[Translation done.]